

CHANGES IN OXIDATION STATE OF CHROMIUM DURING LDEF EXPOSURE

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INTRODUCTION

The solar collector used for the McDonnell-Douglas Cascade Variable Heat Pipe, Experiment A0076 (Michael Grote - Principal Investigator) was finished with black chromium plating as a thermal control coating. The coating is metallic for low emittance, and is finely microcrystalline to a dimension which yields its high absorptivity. An underplate of nickel was applied to the aluminum absorber plate in order to achieve optimal absorptance characteristics from the black chromium plate surface.

Experiment A0076 was located at tray position F9, receiving a projected 8.7×10^{21} atomic oxygen atoms/sq.cm and 11,200 ESH solar radiation. During retrieval, it was observed that the aluminized kapton thermal blankets covering most of the tray had been severely eroded by atomic oxygen, and that a "flap" of aluminum foil was overlaying a roughly triangular shaped portion of the absorber panel (see figure 1). The aluminum foil "flap" was lost sometime between LDEF retrieval and deintegration. At deintegration, the black chromium was observed to have discolored where it had been covered by the foil "flap" (see figure 2). The following is a summary of the investigation into the cause of the discoloration.

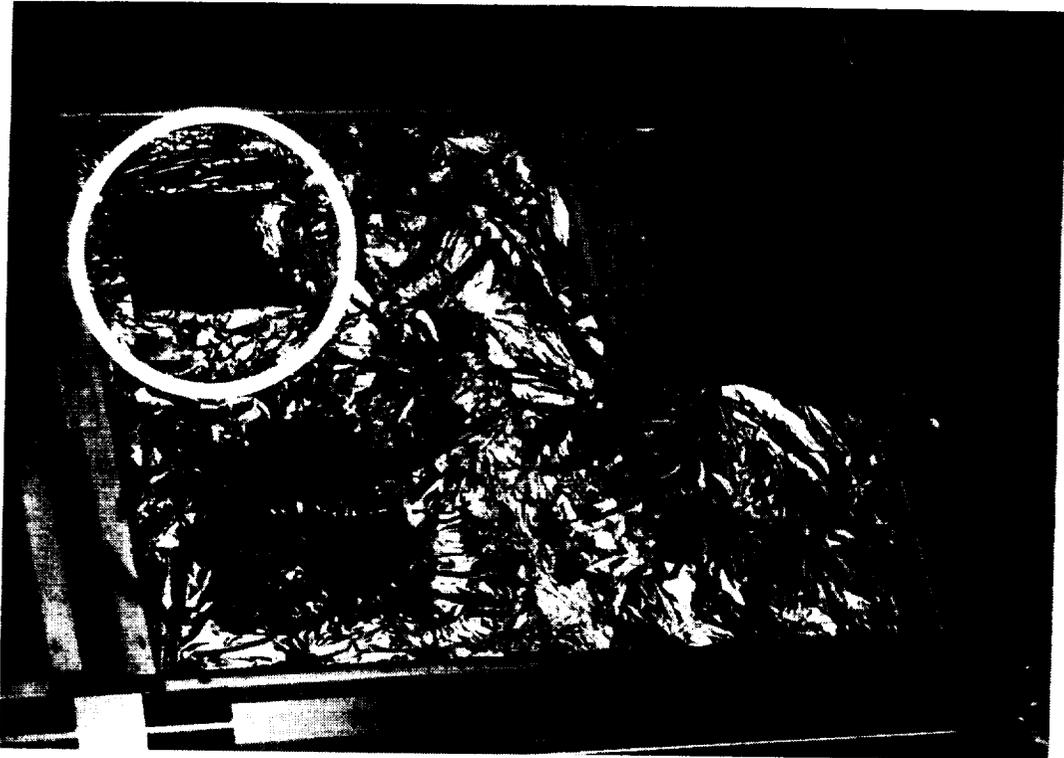


Figure 1. On-Orbit Photograph of Experiment A0076.

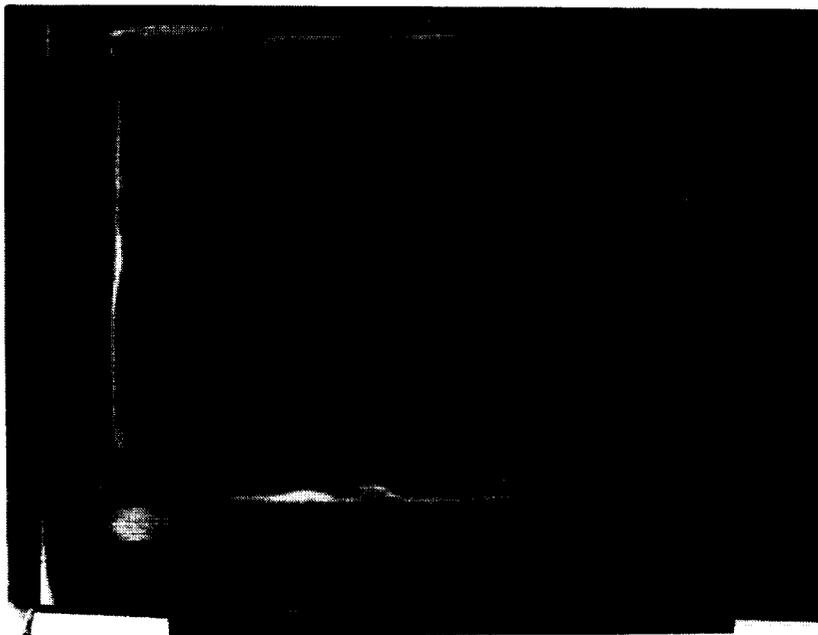


Figure 2. Close-Up on Solar Collector Panel After Deintegration.

Optical properties of the black chromium surface were measured and results are shown in figures 3 and 4. Measurements made at unexposed areas of the absorber panel indicate that these areas still meet or exceed the coating specification performance criteria ($\alpha > 0.90$, $\epsilon < 0.10$). Surfaces exposed to atomic oxygen and UV radiation for the full LDEF mission now have a blue tint. The optical properties of the blue area were minimally affected with a slight reduction in absorptance and no change in emittance. However, the surface which was covered with the aluminum flap for an unknown portion of the mission had degraded significantly in absorptance and slightly in emittance, resulting in a tan-color appearance.

The original hypothesis for this discoloration effect was based on contamination, in which the kapton film decomposition products from its atomic oxygen erosion would be deposited onto the black chromium surface. IR spectroscopy of specimens taken from the discolored area did not yield any measurable absorptions by organic contamination, however.

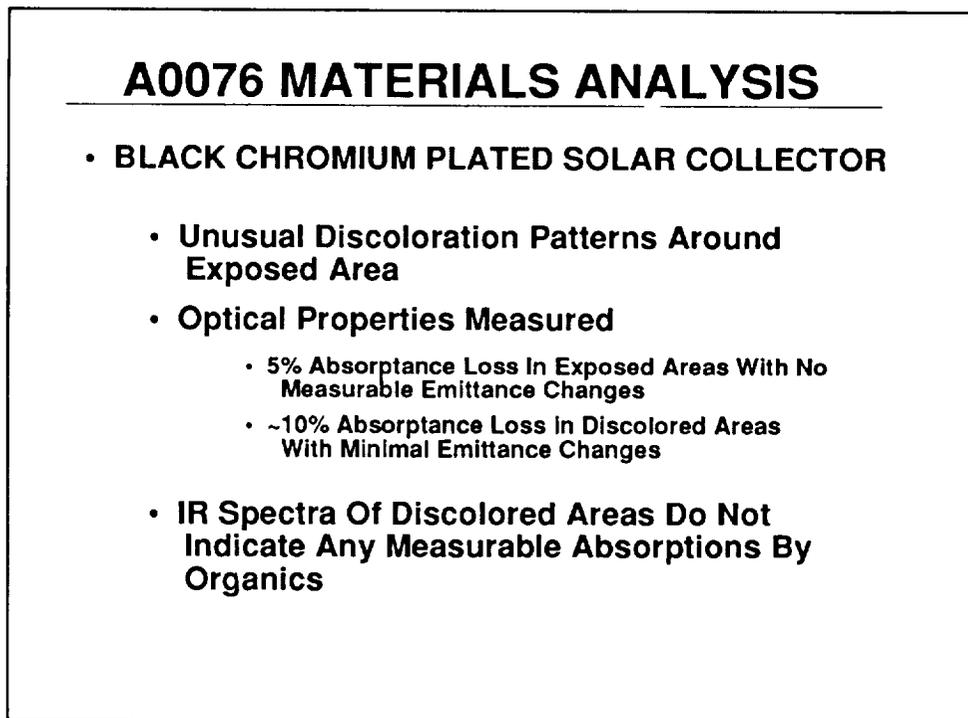
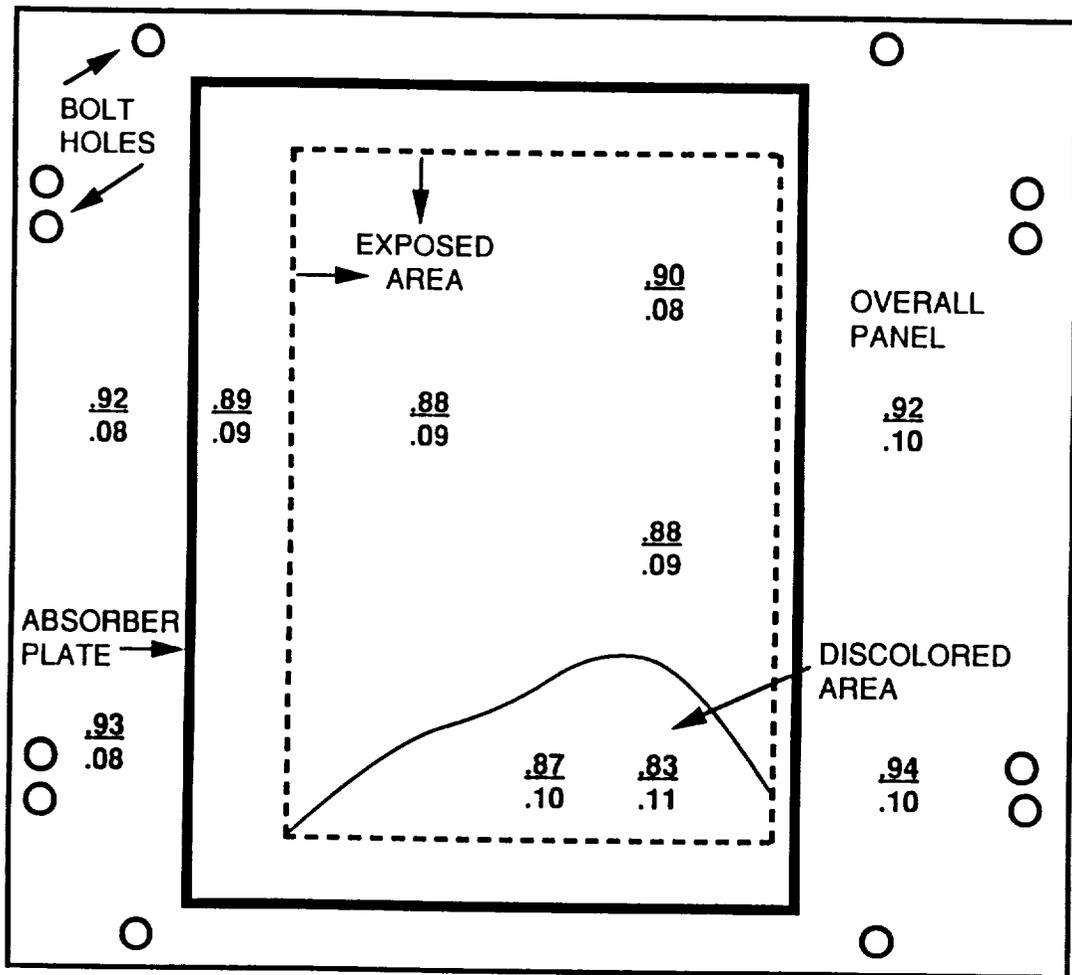


Figure 3.

McDonnell Douglas (LDEF F9 / A0076) Solar Collector
 Black Chrome Oxide Plated 7075 Aluminum



Readings are absorptance
emittance

Figure 4.

X-ray mapping was conducted for specimens taken from the discolored area. Silicon contamination was observed, but this contamination was limited to the extreme corners of the exposed absorber panel area. Silicone contamination was also observed on other structural parts from the interior of the A0076 experiment (fig. 5).

EDX was performed, an example of which is shown in figure 6. The penetration depth of the EDX analysis permits detection of the nickel underplate through the thin chromium layer. No discernable differences between nominal and discolored areas were detected.

SEM of the unexposed, exposed, and exposed and discolored areas of the black chromium are shown in figure 7, at 10,000X magnification. These views indicate fewer and more rounded crystallites in the discolored areas, but the same can be said for the exposed area which did not change significantly in optical properties.

Auger emission spectroscopy profiles were made of the three different areas; these are shown in figures 8, 9, and 10. Differences between the spectra are slight, and the elemental profiles are broad, making interpretation difficult. It appears that the chromium layer has thickened and the oxygen to chromium ratio has increased for the discolored (tan) area, as one compares spectra to those for the blue and then black areas.

A0076 MATERIALS ANALYSIS

- **BLACK CHROMIUM PLATED SOLAR COLLECTOR
(Continued)**
 - **X-Ray Mapping Indicates Silicon Contamination Localized To Extreme Corners**
 - **EDX Detects Nickel Underplating**
 - **SEM Indicates Fewer And Rounded Crystallites In The Discolored Area**
 - **Surface Spectroscopy Measurements Indicate A Thickening Of The Surface Oxide In The Discolored Area**

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Figure 5.

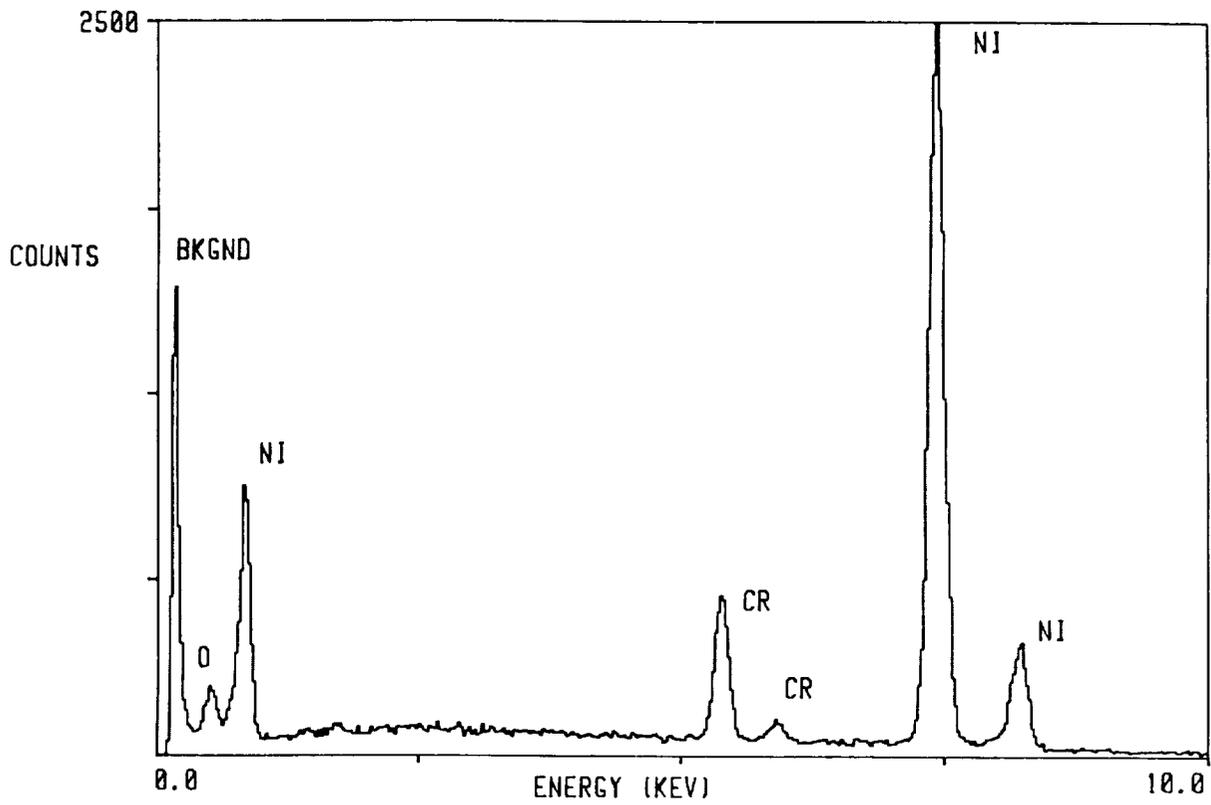
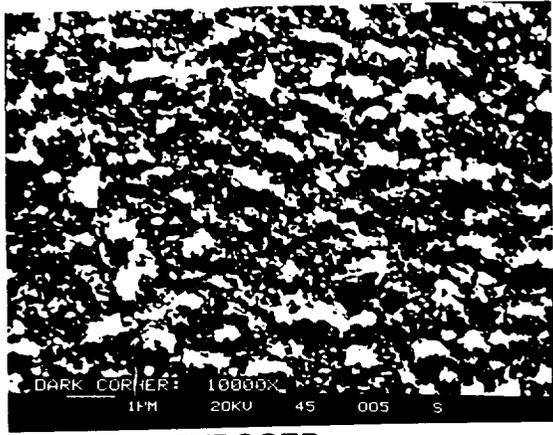
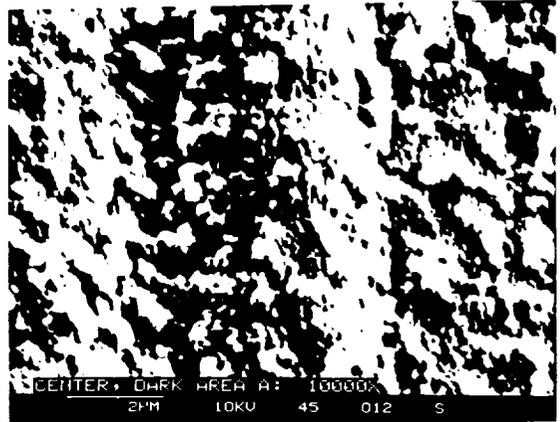


Figure 6. EDX of Discolored Area on Solar Collector Panel.

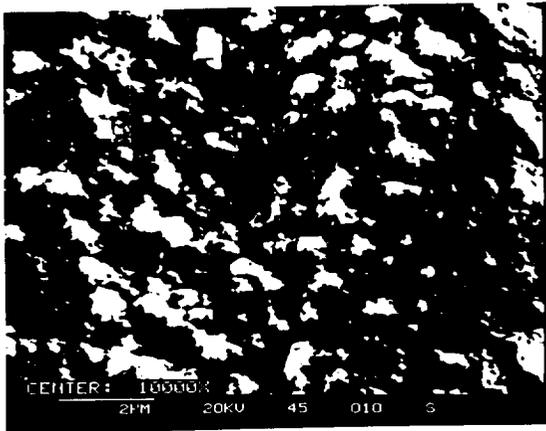


UNEXPOSED

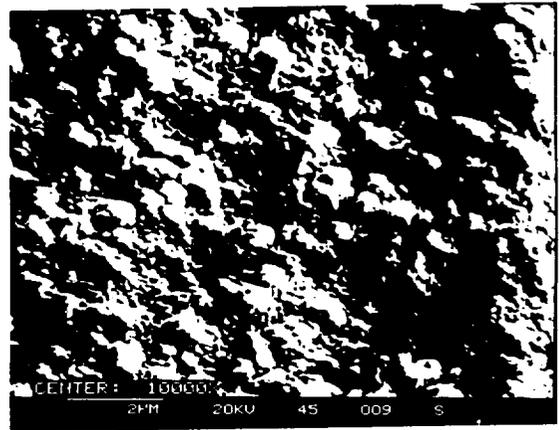
B



EXPOSED AND DISCOLORED



EXPOSED



EXPOSED AND DISCOLORED

Figure 7. SEM of Black Chromium Surfaces.

(Original photographs unavailable).

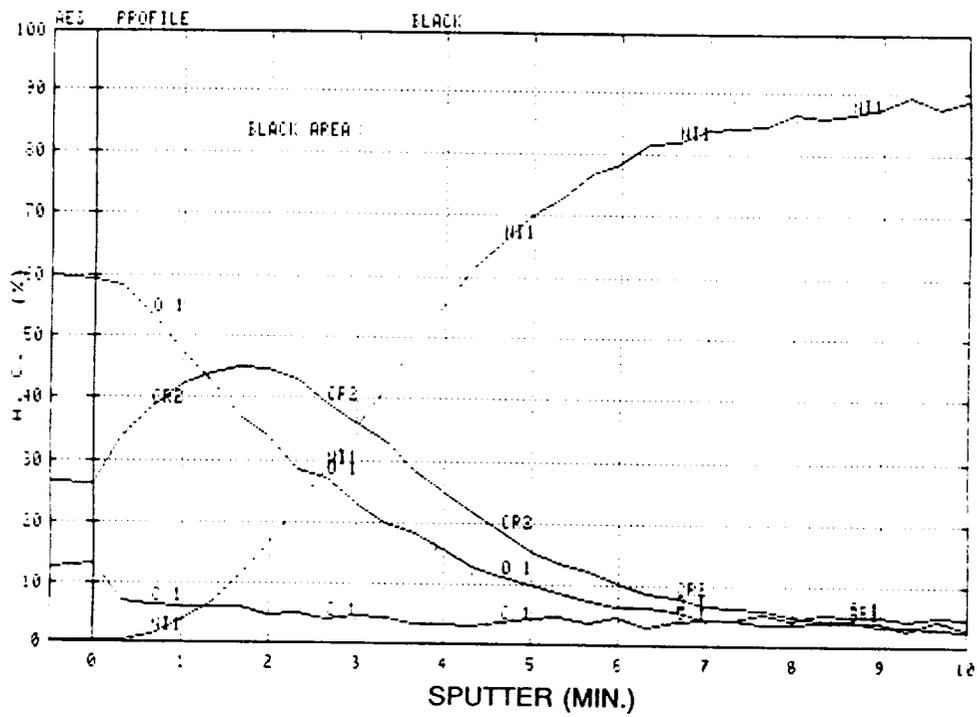


Figure 8. Auger Emission Spectroscopy Profile of Unexposed (Black) Area.

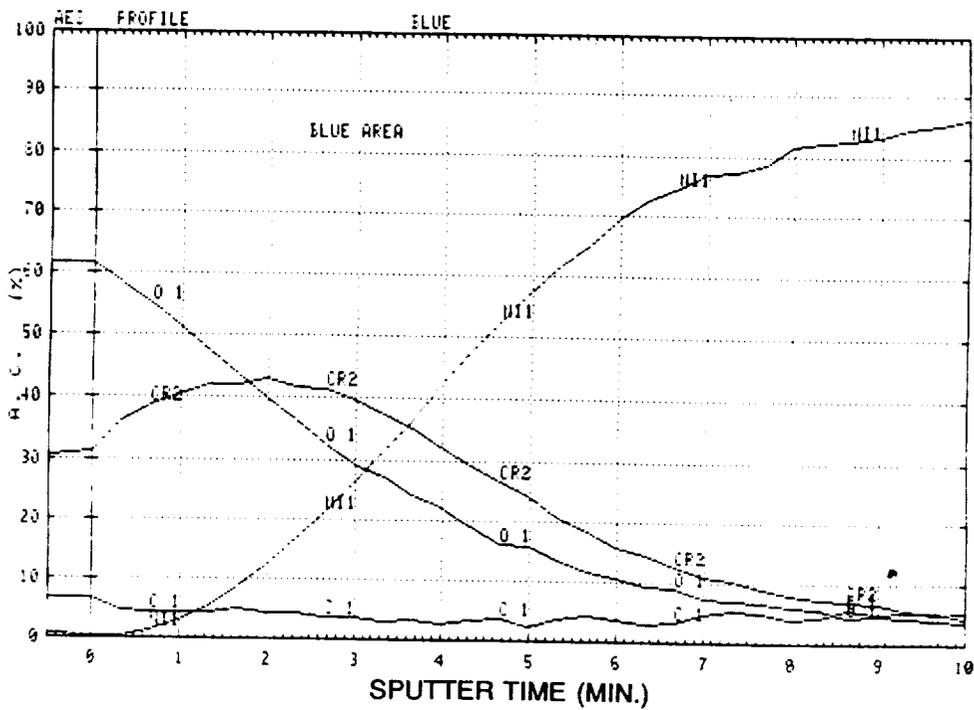


Figure 9. Auger Emission Spectroscopy Profile of Exposed (Blue) Area.

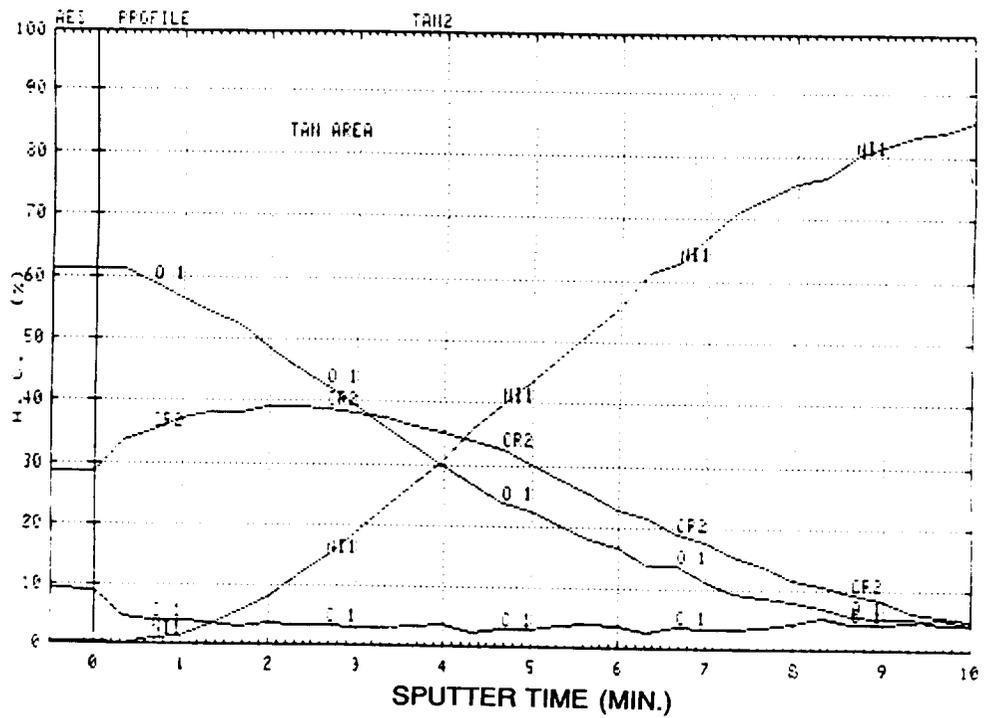


Figure 10. Auger Emission Spectroscopy Profile of Exposed & Discolored (Tan) Area.

These results are not conclusive. But based on the Auger results the following hypothesis is proposed. The discoloration was induced by a thermal effect, caused by the close proximity of the aluminum foil flap to the black chromium coating. The aluminum foil flap, with a low thermal mass and high absorptance to emittance ratio, became very hot, accelerating the atomic oxygen driven oxidation of the chromium. This hypothesis is currently in test using ESCA profiling to determine the chromium oxidation states as a function of depth (fig. 11).

A0076 MATERIALS ANALYSIS

HYPOTHESIS

- **Discoloration Was Caused By A Heating Effect. Residual Aluminum Foil (Low Thermal Mass, High a/e) From Degraded Thermal Blankets Super-Heated Areas Where It Contacted The Black Chromium Coating. Oxidation Of The Chromium Was Accelerated Due To Increased Temperature.**

Figure 11.